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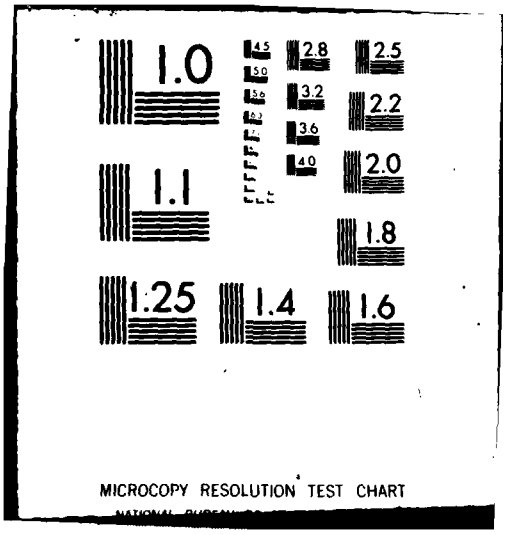
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Tetrafluoroethylene. A Convenient Laboratory Preparation

by

Ronald J. Hunadi and Kurt Baum

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Fluorochem, Inc.
680 S. Ayon Ave.
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A convenient laboratory preparation of tetrafluoroethylene was developed. Polytetrafluoroethylene was heated in a quartz flask at 600-650°C under vacuum. Tetrafluoroethylene was generated rapidly and was trapped with liquid nitrogen.

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Tetrafluoroethylene. A Convenient Laboratory Preparation[1]

Ronald J. Hunadi, Kurt Baum*

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Lewis and Naylor reported in 1947[2] that polytetrafluoroethylene underwent fragmentation at 600-700°C and that the product composition was dependent upon the reaction pressure. At pressures greater than 150 mm, tetrafluoroethylene, hexafluoropropene and octafluorocyclobutane were obtained. Pyrolysis at pressures between 40 and 70 mm produced tetrafluoroethylene and hexafluoropropene, whereas only tetrafluoroethylene was obtained at pressures below 5 mm. Madorsky, et al. subsequently reported similar results, and studied the reaction in detail at lower pyrolysis temperatures.[3] The ready availability of polytetrafluoroethylene at this time makes this reaction attractive as a laboratory source of tetrafluoroethylene.

We have utilized the vacuum pyrolysis of polytetrafluoroethylene with simple apparatus for the preparative scale generation of the monomer. Polytetrafluoroethylene powder was heated in a quartz flask at a pressure of 0.6 to 2 mm Hg. The flask was heated by means of a top-opening box furnace, maintained at 600-650°C (air temperature), and the reaction was complete in 0.5 h. The monomer, condensed in a liquid-nitrogen-cooled receiver, was obtained in 90-96% yield, and no impurities other than entrained tetrafluoroethylene powder were detected. The tetrafluoroethylene yield can be determined conveniently by measuring the liquid volume at -100°C. For applications where the amount used is not critical, the material can be transferred to a reaction vessel

directly from the liquid nitrogen trap. A 200 mL quartz flask was convenient for the generation of 15 to 80 g of tetrafluoroethylene. The product was used for the preparation α,ω-diiodoperfluoroalkanes by the iodine telomerization reaction.[4]

Experimental

Tetrafluoroethylene. A quartz long-necked 24/40 200 mL round bottom flask was loaded with 15.0 g of polytetrafluoroethylene and was connected with a pyrex adapter to a vacuum trap. The system was purged with nitrogen and was evacuated to 0.6 mm Hg. The trap was cooled with liquid nitrogen and the quartz flask was heated with a top-opening box furnace that had been preheated to 650°C (air temperature). Heating was maintained at 630-650°C and the pressure at 0.6 to 2 mm as the depolymerization progressed. The reaction was complete in 30 min. The liquid nitrogen trap was removed and the product was condensed into a calibrated tube (maintained at -100°C) to give 9.5 mL (96%) of tetrafluoroethylene, bp -76°C, lit.[5] -76.5°C.

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1. This work was supported by the Office of Naval Research.
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